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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant(s): Donald R. Huffman et al. Examiner: P. DiMauro

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Docket: 7913zazy

For: NEW FORM OF CARBON

Assistant Commissioner for Patents  
Washington, DC 20231

DECLARATION OF DONALD R. HUFFMAN PURSUANT TO  
37 C.F.R. §1.131

I, DONALD R. HUFFMAN, declare and say as follows:

1. I am an applicant of the above-identified application.
2. The invention disclosed and claimed in the above-identified application was completed in the United States of America prior to August 23, 1990, the publication date of Russian Patent No. SU 1587200 (hereinafter "Russian Patent").
3. It is my understanding that the Russian patent is being cited to show that it was known prior to the filing date of the above-identified application that  $C_{60}$  is soluble in toluol, a non-polar solvent. It is also my understanding that the USPTO has taken the position that in view of the teachings of the Russian patent, it would have been obvious to extract  $C_{60}$  from a sooty carbon product with a non-polar solvent.
4. An aspect of the present invention is directed to a process for forming fullerenes, including  $C_{60}$  and/or  $C_{70}$  in macroscopic amounts, which comprises (a) vaporizing a carbon source in the presence of an inert quenching gas under conditions effective to form a sooty carbon product comprising fullerenes, said fullerenes being present in said sooty carbon product in amounts capable of extracting the fullerenes therefrom in macroscopic amounts and (b) extracting the fullerenes from said sooty carbon product.

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5. As described in the application, one method of separating fullerenes from the sooty carbon product is to use a non-polar solvent, such as benzene, to extract the fullerenes from the sooty carbon product.

6. The preparation of fullerenes including the physical separation of fullerenes from the sooty carbon product utilizing a non-polar solvent was effected in my laboratory in the Physics Department at the University of Arizona prior to the publication date of the Russian Patent.

7. As evidence thereof, annexed hereto and made a part hereof are Exhibits A and B consisting of photocopies of various laboratory records generated in the laboratories at the University of Arizona in relation to the subject matter described in Paragraphs 4 and 5. These data describe the process of preparing a representative embodiment of the present invention,  $C_{60}$ , in which the  $C_{60}$  was extracted from the sooty carbon product with a non-polar solvent, such as benzene.

8. The invention was completed by me or by scientists and/or technicians working under my direct supervision or control prior to August 23, 1990. Data and information not pertinent to this embodiment and dates have been masked out in the preparation of these photocopies.

9. Exhibit A, consisting of 3 pages, illustrates the isolation of a representative embodiment of the present invention,  $C_{60}$ , from the sooty carbon product. The sooty carbon product, identified in the document as "smoke", was produced by the vaporization of graphite in the presence of helium, as described in the above-identified application and more particularly in Example 1 on Page 16 thereof.

10. As indicated on Page A-1 in Exhibit A,  $C_{60}$  was extracted and separated from the sooty carbon product, referred

to therein as "smoke", with a non-polar solvent, such as benzene.

11. More specifically, Exhibit A indicates that the "smoke" was mixed together with benzene. The material soluble in the benzene, i.e., the dark red liquid, was isolated and separated from that which is insoluble therein. The benzene fraction was collected and vaporized to produce a golden residue.

12. The identity of the product isolated in Paragraph 11 was verified by UV data as well as IR data, X-ray crystallography, mass spectra, among other tests.

13. Exhibit A-3 is the UV spectra of the  $C_{60}$  product described in Paragraph 11 that was further purified by sublimation.

14. Exhibit B consists of 1 page. It indicates that various other non-polar solvents, such as carbon disulfide and carbon tetrachloride could be utilized instead of benzene to extract the  $C_{60}$  from the soot.

15. Moreover, Exhibit B shows that polar solvents, such as water, acetone, ethanol, methanol and propanol could not be used to extract the  $C_{60}$ .

16. Thus, the present invention reciting the use of non-polar solvents, such as benzene, for the extraction of fullerenes, such as  $C_{60}$ , from the sooty carbon product produced from the vaporization of a carbon source in the presence of an inert gas was completed in the laboratories at the University of Arizona, Tuscon, Arizona prior to August 23, 1990, the publication date of the Russian Patent.

17. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that

willful false statements and the like so make are punishable by fine or imprisonment or both under section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

May 3, 1996  
Date

Donald R. Huffman  
Donald R. Huffman

REDACTED

Scrape smoke off chimney + sides of chamber.  
Combine smoke + benzene in a test tube. Stir thoroughly.

Centrifuge for ~ 10 min.

Pour Dark red liquid into quartz crucible -  
place crucible on warm hot plate (cool enough to  
be touched for a few seconds) and heat until all  
benzene has evaporated. A brownish golden residue  
should remain.

REDACTED

Removing volatiles from C<sub>60</sub> samples

REDACTED

2.0

Abs

0.7

0.6

0.5

Abs

0.3

0.1

0.0

200

Wavenumber

200

A-3

B

REDACTED

solvent for  $\text{C}_{60}$ . Success was  $\text{CS}_2$  and  $\text{CCl}_4$  with a moderate  
offered various for benzene. Factors included water,  
acetone, ethyl, methanol, propanol.

B-1



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